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<b>(54) Title:</b> DIAMOND-LIKE CARBON COATED TRANSDUCERS FOR MAGNETIC RECORDING MEDIA		
<b>(57) Abstract</b>		
<p>A method is provided for manufacturing a diamond-like carbon coated magnetic transducer assembly with superior wear resistance, and improved lifetime. The invention discloses a magnetic transducer with a composite coating structure including an adhesion-enhancing a first interlayer containing silicon and an ion beam deposited DLC top layer. According to the method, the surface of the magnetic transducer substrate surface (3) is bombarded with energetic ions from an ion source (4) to preform sputter etching, a silicon containing interlayer is deposited utilizing an ion beam source (7) and a target (10) to sputter deposit the interlayer, and following completion of the deposition of the interlayer a DLC top layer is deposited by ion beam deposition.</p>		

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## DIAMOND-LIKE CARBON COATED TRANSDUCERS FOR MAGNETIC RECORDING MEDIA

### Field of the Invention

5 This invention relates to transducer assemblies utilized in magnetic recording devices. More particularly, the invention relates to a transducer for use with magnetic recording media *e.g.* thin film magnetic heads, magnetoresistive (MR) read heads, inductive heads, sliders, and tape heads.

### Background of the Invention

10 Amorphous diamond-like carbon (DLC) films are so-named because their properties resemble, but do not duplicate, those of diamond. Some of these properties are high hardness (HV = about 1,000 to about 5,000 kg/mm<sup>2</sup>), low friction coefficient (approximately 0.1) and transparency across the majority of the electromagnetic spectrum. At least some of the carbon atoms in DLC are bonded  
15 in chemical structures similar to that of diamond, but without long range crystal order. Although the term DLC was initially intended to define a pure carbon material, the term DLC now used includes amorphous, hard carbon materials containing up to 50 atomic percent of hydrogen. Other names for these hydrogen-containing DLC materials are "amorphous hydrogenated carbon",  
20 hydrogenated diamond-like carbon, or diamond-like hydrocarbon. The structure of these hydrogen-containing hard carbon materials may be best described as a random covalent network of graphitic-type structures interconnected by sp<sup>3</sup> linkages, although the definitive structure of the films has yet to be universally accepted. In keeping with the majority of the previous literature and art, the term  
25 D.L.C is used in the present invention to refer to both the amorphous non-hydrogenated hard carbon materials, and the amorphous hydrogenated hard carbon materials.

Many methods for directly depositing DLC films are known in the prior art, including (i) direct ion beam deposition, dual ion beam deposition, glow discharge,  
30 radio frequency (RF) plasma, direct current (DC) plasma or microwave plasma deposition from a carbon-containing gas or vapor which can also be mixed with hydrogen and/or inert gas, (ii) electron beam evaporation, ion-assisted evaporation,

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magnetron sputtering, ion beam sputtering, or ion-assisted sputter deposition from a solid carbon target material, or (iii) combinations of (i) and (ii).

DLC films are well known in the art and have been recognized as potential coatings to enhance the abrasion resistance of various substrate materials, including recording media. The DLC coatings possess excellent mechanical properties such as high hardness and low coefficient of friction, and exhibit excellent resistance to abrasion and chemical attack by nearly all known solvents, bases, and acids. However, it has been found that the DLC coatings will impart improved wear resistance to the substrate only if the adherence of the coating to the parent substrate is excellent.

The most obvious and common approach to coating a substrate is to apply the DLC coating directly onto a clean surface which is free of residue. However, this approach often results in a DLC coating which displays inadequate adhesion, and therefore, poor wear resistance. DLC coatings are typically under significant compressive stress, on the order of  $5 \times 10^9$  to approximately  $5 \times 10^{10}$  dynes/cm<sup>2</sup>. This stress greatly affects the ability of the coating to remain adherent to the substrate. Additionally, the surface of the substrate to be coated often contains alkali metals, oxides, and other contaminants which can inhibit bonding of the DLC coating. Therefore, less obvious methods are required to produce a substrate with a highly adherent DLC coating which provides excellent abrasion resistance.

Magnetic transducers which include thin film magnetic heads, magnetoresistive (MR) read heads, inductive heads, sliders, and tape heads, utilized in magnetic recording media have been known in the art for many years. Their susceptibility to damage is also well known. One example of a magnetic transducer used in recording media is a magnetic head slider. The slider supports a thin film magnetic read/write head, which is formed by depositing layers of magnetic material, electrically conductive material, and electrically insulating material to form the magnetic pole pieces and magnetic gap which are necessary for the transducing function with the magnetic coating on a magnetic recording medium. After lapping the magnetic head to a predetermined "throat height" dimension, a

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pattern of rails is produced on the lapped surface to form an air bearing surface which is used to "fly" the magnetic head over the magnetic recording medium.

During operation, a magnetic head slider typically flies with its air bearing surface less than a few microinches (1 microinch = 254 Å) above the magnetic recording medium. The air bearing surface of the slider contacts the magnetic disk during start-up and shutdown of the disk rotation, and sometimes inadvertently during operation. In conventional sliders and recording media, this contact results in transfer of disk material (wear debris) to the slider, which degrades the aerodynamics of the slider and increases friction. The presence of the wear debris and the increased friction can at times result in catastrophic failure and loss of stored information.

Tape heads used with magnetic recording tape also suffer failure due to wear and corrosion. In this case, the magnetic pole pieces are worn away by abrasive materials in magnetic tape. The magnetic materials on the transducer are also degraded by environmental corrosion.

In addition to mechanical wear, magnetic transducers such as thin film magnetic heads and magnetoresistive heads are fabricated from materials that are attacked by atmospheric constituents, such as moisture. Prolonged exposure of the transducer materials to atmosphere often results in degradation of performance due to oxidation and corrosion of the head materials.

Protective DLC overcoats for thin film metal alloy disks are well known. A review of the field was presented by H. Tsai and D. Bogy in "Characterization of Diamondlike Carbon Films and Their Application as Overcoats on Thin-film Media for Magnetic Recording", J. Vac. Sci. Tech. A5 (1987) 3287-3312. Illustrative from the prior art are the following references:

Aine, U.S. Pat. No. Re 32,464 teaches a magnetic recording medium coated with a sputter-deposited graphitic carbon protective layer having thickness between 1-5 microinches. Use of an adhesion-promoting carbide-forming layer between the disk and the carbon film is disclosed and claimed. Additionally, "the magnetic transducer head portion which occasionally sinks into contact with the recording medium is preferably formed of or coated with carbon, preferably in the form of graphite, to provide a low friction wear resistant contacting surface with the

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recording medium". The carbon layer may also be deposited by ion plating. Aine further states that the sputtering apparatus allows the substrate surfaces and the surfaces of subsequent layers which are to be coated to be cleaned by bombarding such surfaces with ions for cleaning away any surface contaminants.

5 Michihide et al., EP 216 079 A1, teach a method for manufacturing a thin carbon film on the surface of a sliding member such as a hard disk of a magnetic recording device which contacts another member, in which the carbon thin film is formed by sputter deposition from a glassy carbon sputtering target. Triode direct current sputtering, diode radio frequency glow discharge sputtering and magnetron  
10 sputtering are the methods disclosed for sputter deposition using the glassy carbon target material.

Howard, U.S. Pat. No. 4,778,582, describes a prior art sputter deposition technique for manufacture of a thin film magnetic disk coated with amorphous hydrogenated carbon. In this method, amorphous hydrogenated carbon is  
15 deposited by sputtering a carbon target in an atmosphere of argon and hydrogen. The deposition rate is approximately 6-7 Å/minute. A 100 Å thick titanium layer is used as an adhesion layer.

Meyerson et al., U.S. Pat. No. 4,647,494, disclose an improved wear-resistant coating for metallic magnetic recording layers, where the improved  
20 coating is a non-graphitic hard carbon layer strongly bound to the underlying metallic magnetic recording layer by an intermediate layer of silicon, having a thickness less than 500 Å. The silicon layer can be as thin as a few atomic layers. The hard carbon layer has a thickness in the range of about 25 Å to 1 micron. The preferred method for depositing both the silicon intermediate layer and the hard  
25 carbon layer is claimed to be plasma deposition. The patentees further teach that the silicon interfacial layer can be deposited by any known technique, e.g. evaporation, sputtering, or plasma deposition. Plasma deposition is preferred because the same processing equipment and steps can be used to form the silicon layer and the overlying hard carbon layer. Therefore, both the silicon layer and the  
30 hard carbon layer can be formed by plasma deposition without breaking the vacuum in the system. Changing the source gasses, e.g., from silane to acetylene, are the only additional steps that are necessary. The patentees also state that hard

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carbon layer can be also deposited by sputtering and other methods. The patentees still further teach the silicon layer is exposed for 45 minutes to a hydrogen plasma to provide both reactive and sputtered cleaning of the growth surface and also to further reduce the native oxide layer on the silicon. This cleaning time is very  
5 long, and unacceptable for industrial production. It is further stated that the hydrogen plasma preclean does not, by itself completely remove the native oxide from the silicon substrate. Thus, improved methods of precleaning the substrate prior to depositing the hard carbon coating are needed. The patentees also disclose difficulties in the use of plasma deposition of the hard carbon layer; see column 6,  
10 line 67 through column 7, line 3.

Japanese Laid Open Pat. Application (Kokai) No. 1-287819, Shinora, claims a magnetic recording medium (magnetic disk or magnetic tape) in which a diamond-form hard carbon thin film is located on a strongly magnetic metal thin film with a silicon or germanium layer, interposed between the two. Ion plating,  
15 high frequency sputtering, and electron beam evaporation are disclosed as methods for depositing silicon and germanium films, from thickness of 20 to 50 Å. The diamond-like carbon film has a thickness between 50 and 100 Å and can be formed by high frequency sputtering, ion beam deposition, or plasma acceleration. The diamond-like carbon layer is then overcoated with a lubricant to form the final  
20 product.

Endo et al., U.S. Pat. No. 4,774,130, discloses a magnetic recording medium composed of a magnetic film formed on the surface of a disk-shaped substrate and a protective film further formed on the surface of the magnetic film. The protective film is composed of a first layer containing silicon, germanium, or  
25 chromium oxide, and a second (top) layer of amorphous carbon or graphite-containing amorphous carbon. The first and second layers can be formed by a variety of sputtering techniques including magnetron sputtering, diode sputtering, and ion beam sputtering. The patentees teach that the first layer containing chromium oxide, silicon, and germanium formed on the magnetic film  
30 excels in resistance to corrosion and weather conditions. The second layer of amorphous carbon or graphite-containing amorphous carbon excels in lubricating property. The thickness of a first layer of elemental silicon is in the range of 100

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to 300 Å. It is also taught that the second (carbon) layer is desired to have a thickness in the range of 200 to 700 Å. If the thickness is smaller than 200 Å, the effectiveness of the second (carbon) layer in improving the lubricating property is not sufficient. The patentees further state that if the resistance of the carbon layer exceeds  $10^4$  ohm-cm, the amorphous carbon assumes a texture approximating the diamond structure and shows insufficient lubricating properties. In their examples, the patentees demonstrate that carbon films having properties which are more diamond-like than those in the claimed range perform much worse in contact-stop-start (CSS) tests than the amorphous carbon or graphite-containing amorphous carbon films of the patentees' invention. For example, the graphite-containing amorphous carbon layers provide resistance for 40,000-130,000 CSS cycles, whereas carbon films with the diamond-like structure provide resistance for 10,000-16,000 CSS cycles. In addition, it is shown that the graphite-containing amorphous carbon films are much more resistant to permeation by water than with the diamond-like structure.

Kurokawa et al., U.S. Pat. No. 4,717,622, discloses a magnetic recording medium with a protective layer of high hardness carbon synthesized under a low temperature and low pressure gas plasma. The magnetic recording medium is useful in a system where the magnetic head contacts the magnetic recording medium. The diamond-like carbon film has a Vicker's hardness of more than  $2,000 \text{ kg/mm}^2$ , and a specific resistance of  $10^7$  ohm-cm to  $10^{13}$  ohm-cm. The patentees refer to their diamond-like carbon coatings having properties in the aforementioned range as "amorphous diamond". It is specifically stated that amorphous carbon films having a hardness in the range of  $1,500 \text{ kg/mm}^2$  are "soft". These "soft" carbon films are outside the critical teaching of this reference.

Nakamura et al., U.S. Pat. No. 4,804,590, teach an abrasion resistant magnetic recording member comprising a carbonaceous surface protective film on a surface of a magnetic film on the surface of a nonmagnetic substrate. The protective film has a lower layer of comparatively hard carbonaceous film and an upper layer of comparatively soft carbonaceous film. An intermediate layer of chromium, titanium, etc. may be used to improve the adhesion of the carbonaceous film to the magnetic film. The lower carbonaceous layer contains 5 atomic percent

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or less of hydrogen, fluorine, or a combination of hydrogen and fluorine, and the upper carbonaceous layer contains 6 atomic percent or more of hydrogen, fluorine, or a combination of hydrogen and fluorine. The lower carbonaceous layer may be a sputtered carbonaceous film, and the upper layer may be a plasma chemical  
5 vapor deposited (PCVD) carbonaceous film.

Additionally, protective DLC coatings on magnetic head sliders have been discussed in the prior art. Illustrative are the following references:

IBM Technical Disclosure Bulletin, Vol. 25, No. 7A (1982) page 3173 discusses a magnetic slider made of silicon which is coated with an extremely hard  
10 surface layer of silicon carbide or diamond-like carbon. The sufficient thickness of the silicon carbide or diamond-like carbon surface layer is in the range of 50 nm to 100 nm. The diamond-like carbon layer can be produced by ion beam or plasma deposition.

Bleich et al., U.S. Pat. No. 5,151,294, discloses a method for depositing a  
15 thin protective carbon film on the air bearing surface of a slider in a magnetic recording disk file by contacting the slider and a rotating magnetic disk for a time sufficient to cause transfer of the carbon from a carbon overcoated disk to the air bearing surface of the slider. The patentees teach that the carbon film formed on the slider is an essentially amorphous hydrogenated carbon film approximately 50  
20 Å in thickness.

Japanese Laid Open Pat. Application (Kokai) No. 3-25716, discloses a magnetic head slider, characterized by the fact the head slider body is made of a soft ceramic material, such as ferrite, and a hard ceramic membrane such as carbon, silicon, zirconium dioxide, aluminum oxide, or the like, which is bonded  
25 and molded onto at least the pressure-receiving surface of the slider which faces the magnetic disk. This disclosure is said to offer advantages in manufacturing of the slider, because softer ceramic substrate materials are used in processing. The hard ceramic membrane has a thickness in range of several hundred Angstroms, and may be made by a plasma chemical vapor deposition (PCVD) method.

30 Head et al., U.S. Pat. No. 4,130,847, describes a magnetic head slider having a protective coating preferably chromium over at least the magnetic head.

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The coating is produced in a recess within the slider body to a thickness as small as 10 microinches.

Grill et al., U.S. Pat. No. 5,159,508, teaches a magnetic head slider coated with an adhesion layer and a protective diamond-like carbon layer. The coating is fabricated onto the substrate after a lapping operation, but before patterning of the rails onto the slider, which protects the magnetic head during the fabrication process. The slider has at least two rails on the air bearing surface, and the rails have a protective coating comprising an adhesion layer, typically about 10 Å to 50 Å in thickness, and a thin layer of amorphous hydrogenated carbon, less than about 250 Å thick. Grill et al. teach that the two layers of the protective coating can be deposited by any suitable technique, e.g. PACVD, ion beam or laser techniques. The preferred technique is by the use of a DC biased substrate in an RF plasma deposition apparatus.

Chang et al., U.S. Pat. No. 5,175,658, claim a magnetic slider similar to that disclosed in Grill, *et al.*, U.S. Pat. No. 5,159,508, discussed above, but with an additional thin masking layer on top of the amorphous hydrogenated carbon layer. DC magnetron sputtering and RF magnetron sputtering are presented as preferred methods for depositing the amorphous hydrogenated carbon film.

Japanese Laid Open Pat. Application JP 58-150,122 describes a magnetic head which has a thin film of a material, e.g. carbon, having a lubricating effect on the surface of the head which faces the magnetic recording medium. The thickness of the film is disclosed to be in within the range of 200 Å to 5000 Å.

German Pat. Application No. DE 3,714,787 describes a storage system in which the surface of a magnetic disk is coated with friction-reducing carbon and the rails of the magnetic head slider are coated with a friction reducing carbon. The thickness of the carbon is 10 to 1000 Å.

Published Pat. Application PCT/US88/00438 discloses a magnetic head slider having a magnetic head which is built within one of the side rails. A wear layer is provided over the slider comprising a 50 Å thick chromium layer and a 200 Å thick carbon layer. Either component of the wear layer can be omitted.

The prior art methods for application of DLC coatings on magnetic media transducers such as thin film heads and tape heads all suffer from one or more of

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the following deficiencies and shortcomings: (i) difficulty in pre-cleaning of substrates prior to deposition; (ii) adhesion of diamond-like carbon coating; (iii) permeation of amorphous carbon films by water vapor and oxygen; (iv) fabrication of coherent, dense coatings which perform well at thicknesses less than 200 Å; (v) 5 poor electrical resistivity of the coating due to the use of low resistivity Ti, Cr, or Si adhesion layers between the transducer and the DLC coating, or a "graphitic", low resistivity DLC layer; (vi) control of DLC coating properties during a deposition run and batch-to-batch variation of DLC coating characteristics; (vii) DLC coating thickness control and reproducibility of thickness; (viii) part-to-part 10 and batch-to-batch control of DLC coating uniformity; (ix) production readiness and ability to scale-up the deposition process for mass production; and (x) difficulty in coating transducer assembly substrates of complex geometry or configuration.

These shortcomings are highlighted in the following review of the two 15 preferred prior art methods for deposition of DLC coatings on magnetic media and magnetic transducers: RF plasma deposition from a hydrocarbon gas, and magnetron sputter deposition from a carbon target using argon gas, or an argon/hydrogen gas atmosphere. The first problem encountered by both methods is the difficulty in pre-cleaning the substrates prior to deposition of the adhesion 20 layer or carbon film. Typically substrates are pre-cleaned in an inert gas or hydrogen glow discharge (plasma) prior to deposition. This pre-cleaning technique suffers from low cleaning rate, and re-contamination of the substrate by sputtered contaminants which are deposited back onto the substrate.

It has been found that an adhesion-promoting interlayer is required to 25 achieve excellent adhesion between the magnetic transducer substrate and the protective DLC coating. In the prior art, layers such as Ti, Cr, and Si, deposited by magnetron sputter deposition, evaporation, and plasma deposition have been used for this purpose. Achieving a highly dense (non-porous), adherent, adhesion-promoting layer still remains a problem with the prior art techniques. 30 Deposition of a silicon interfacial layer by plasma deposition such as disclosed in U.S. Pat. No. 4,647,494 as discussed above, has the additional disadvantage that the feed gas used is silane, which is an extremely flammable and toxic material.

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Feed gases of this nature are required by the plasma deposition technique for deposition of silicon layers of high purity.

One of the key requirements of the protective DLC coating on magnetic transducers is the need to provide a barrier to moisture and oxygen. This requires  
5 formation of a DLC structure with optimal atom packing density. This atom packing density is maximized by a high degree of ion bombardment during film growth, which is not easily attainable or optimized by the magnetron sputter deposition or plasma deposition methods of the prior art. In addition, the difficulty  
10 in forming DLC films of maximum atom packing density by the magnetron sputter deposition and plasma deposition methods of the prior art makes deposition of coherent, corrosion resistant pinhole-free films less than 200 Å thick extremely difficult.

For many designs of magnetic transducers, it is critical for the protective coating to exhibit high electrical resistivity. This is specially true for  
15 magnetoresistive designs of tape heads or sliders. In these magnetoresistive designs, there are magnetic shields in the heads which shield the magnetoresistive sensor from the other elements of the head. Those shields can be either grounded or at potential, but always at a different potential than the sensor. It is not allowable to provide a path for electrical conduction between the shields to the  
20 sensor. Additionally, it is not allowable to provide an electrically conductive path between the sensor to any other element in head which is maintained at a different electrical potential. Finally, there are situations in which it is deleterious to allow electrical charge to pass directly between the transducer and the recording medium when the transducer and recording medium come into direct contact. An  
25 electrically non-conducting layer can protect against this problem.

Thus, it is often critical for the protective coating to be highly electrically insulating. When a pure argon atmosphere is used, the electrical conductivity of the sputter-deposited carbon film may be too high, causing electrical losses in the transducer. Electrical conduction paths can also be provided by low resistivity Ti,  
30 Cr, or Si adhesion layers of the prior art. Clearly, for many magnetic transducer designs, there is a need for the production of a protective DLC coating in which

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both the DLC layer and the adhesion-promoting interlayer are electrically non-conducting.

Regarding the control of DLC coating properties within a single deposition run, and from batch-to-batch, it is well known that control is difficult with the plasma deposition methods, see Meyerson et al. in U.S. Pat. No. 4,647,494 discussed above. It is known in the prior art that as the hydrogen concentration in hard carbon films increases, the film hardness decreases. Thus, the hard carbon film deposited by the plasma deposition method is not uniform in composition throughout its structure, the top surface of the film being softer than the portion of the film in contact with the silicon layer. Softer DLC layers are disadvantageous from the point of view of higher wear rates.

Because the size and shape of the part to be coated, and its method of fixturing influence the plasma uniformity and plasma density around a part, it is difficult to predict and control deposition thickness uniformity across multiple parts coated within a single coating run using the plasma deposition methods of the prior art.

The deposition rates of amorphous carbon materials by magnetron sputter deposition from a carbon target are very low, on the order of 10 Å/minute or less, see U.S. Pat. No. 4,778,582 discussed above, resulting in excessively long production times. While the plasma deposition methods offer much higher deposition rates, it is difficult to reproducibly control deposition thickness, deposition rate and deposition uniformity across large areas with plasma deposition methods. Due to their inherent high deposition rates, the plasma deposition methods (RF plasma, DC plasma and microwave plasma) are well-suited to fabrication of thick coatings of diamond-like carbon for many industrial applications on wear parts. However, because of the interdependence of process variables such as pressure, gas flow rate, power, and substrate bias, accurate control of deposition thickness is difficult. Thus, it is very difficult to manufacture hard carbon layers as well as silicon adhesion layers with thickness less than 1,000 Å with run-to-run thickness variation of less than approximately 10%. This is a significant disadvantage of the plasma deposition techniques of the prior art for the deposition of DLC coatings on sliders where accurate control of thin (e.g. 200 Å)

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protective layers is required. Very thin ( $< 200 \text{ \AA}$  thick) protective layers is required for ultra-high recording density applications in which the distance between the transducer and the magnetic medium needs to be less than several hundred Angstroms. The thickness of the DLC coating narrows this spacing.

5           Finally, because of the sensitivity of the plasma deposition processes to substrate geometry, it is often impossible to coat parts of complex geometry or configuration. Examples of complex geometry include sliders already mounted on their suspension systems, and tape heads or other transducers which have been packaged complete with electrical connectors (e.g. wires). Thus, the plasma  
10 deposition methods are more conducive to application of the DLC coating while sliders, for example are still joined on a chip as in U.S. Pat. Nos. 5,159,508 and 5,175,658 discussed above.

          An improved manufacturing method would be very desired that allows the DLC coating to be applied to magnetic transducer assemblies of complex geometry.  
15 Additionally, an improved manufacturing method is needed that allows the DLC coating to be deposited as the final step in the transducer manufacturing process. In this way, the DLC coating process could be readily integrated into current transducer assembly manufacturing lines.

          All of the difficulties described above combine to make mass production of  
20 DLC coatings on magnetic transducers by the plasma deposition process and other prior art methods very problematic indeed. Clearly, there is a strong need for an improved method for flexible, reproducible, and high quality mass production of DLC coatings for magnetic transducers.

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### Summary of the Invention

The invention provides a magnetic transducer assembly with superior wear resistance, and improved lifetime. More particularly, this invention provides an ion beam deposited DLC coating to the surface of a magnetic transducer which is  
5 highly adherent, and exhibits greatly improved wear resistance and environmental durability. This invention also provides a low cost and efficient process for mass-producing the diamond-like carbon coated magnetic transducer with improved wear resistance and superior lifetime.

In the present invention, a magnetic transducer is provided with a composite  
10 coating structure including an adhesion-enhancing first interlayer containing silicon and an ion beam deposited DLC top layer. The invention further provides a method for fabricating the protective DLC coating on the magnetic transducer.

In the method of the present invention, after completion of the lapping, or other operation to define the magnetic pole pieces, the surface of the magnetic  
15 transducer substrate is chemically cleaned to remove contaminants, ie. unwanted materials and other contaminants. In the second step, the substrate is inserted into a vacuum chamber, and the air in said chamber is evacuated. In the third step, the substrate surface is bombarded with energetic ions to assist in the removal of residual contaminants, i.e. hydrocarbons and surface oxides, and to activate the  
20 surface. After the substrate surface has been sputter-etched, a silicon-containing interlayer material is deposited, preferably by ion beam sputter deposition. Following completion of the deposition of the interlayer, a DLC outer layer is deposited by ion beam deposition. Once the chosen thickness of the DLC layer has been achieved, the deposition process on the transducer substrates is  
25 terminated, the vacuum chamber pressure is increased to atmospheric pressure, and the DLC-coated magnetic transducer substrates are removed from the vacuum chamber.

### Brief Description of the Drawings

Further features and advantages will become apparent from the following  
30 and more particular description of the preferred embodiment of the invention, as illustrated by the accompanying drawings, in which like reference characters generally refer to the same parts or elements, and in which:

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FIG. 1 is an illustration of the ion beam deposition apparatus used to manufacture DLC-coated magnetic transducers in accordance with the present invention;

FIG. 2A is a graph of frictional force versus the number of test revolutions  
5 in a constant speed drag test for an uncoated  $\text{TiC}/\text{Al}_2\text{O}_3$  slider;

FIG. 2B is a graph of touch down velocity change versus the number of test revolutions in a constant speed drag test for an uncoated  $\text{TiC}/\text{Al}_2\text{O}_3$  slider;

FIG. 2C is a graph of frictional force versus the number of test revolutions in a contact-start-stop test for an uncoated  $\text{TiC}/\text{Al}_2\text{O}_3$  slider;

10 FIG. 2D is a graph of touch down velocity change versus the number of test revolutions in a contact-start-stop test for an uncoated  $\text{TiC}/\text{Al}_2\text{O}_3$  slider;

FIG. 3A is a graph of frictional force versus the number of test revolutions in a constant speed drag test for a slider coated with  $100\text{\AA}$  of DLC according to the present invention;

15 FIG. 3B is a graph of touch down velocity change versus the number of test revolutions in a constant speed drag test for a slider coated with  $100\text{\AA}$  of DLC according to the present invention;

FIG. 4A is a graph of frictional force versus the number of test revolutions in a constant speed drag test for a slider coated with  $50\text{\AA}$  of DLC according to the  
20 present invention;

FIG. 4B is a graph of touch down velocity change versus the number of test revolutions in a constant speed drag test for a slider coated with  $50\text{\AA}$  of DLC according to the present invention;

FIG. 5A is a graph of frictional force versus the number of test revolutions  
25 in a contact-start-stop test for a slider coated with  $100\text{\AA}$  of DLC according to the present invention; and

FIG. 5B is a graph of touch down velocity change versus the number of test revolutions in a contact-start-stop test for a slider coated with  $100\text{\AA}$  of DLC according to the present invention.

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Detailed Description of the Invention

The method of the present invention substantially reduces or eliminates the disadvantages and shortcomings associated with the prior art techniques by providing:

- 5       (1) an improved transducer assembly, e.g. slider, inductive head, magnetoresistive head, tape head, thin film head, and similar devices etc. for use with magnetic recording media, in which the transducer has improved lifetime, resistance to wear, and resistance to corrosion;
- 10       (2) for the manufacture of a transducer assembly for use with magnetic recording media, in which the transducer has improved lifetime, resistance to wear, and resistance to corrosion;
- 15       (3) for the deposition of an amorphous DLC coating onto the surface of a transducer assembly, which surface faces the magnetic recording media, in which the amorphous DLC coating has the properties of high adhesion to the substrate, impermeability to environmental elements such as water vapor and oxygen, high density, and extreme surface smoothness;
- 20       (4) for the deposition of a thin amorphous DLC coating at layer thicknesses as small as 50 Å or less onto the surface of a transducer assembly, which surface faces the magnetic recording media, in which the thin amorphous diamond-like carbon coating provides a protective surface for the transducer;
- 25       (5) for the deposition of a protective amorphous DLC coating onto the surface of a transducer assembly, which surface faces the magnetic recording media, in which the layer thickness and uniformity of the amorphous diamond-like carbon coating are reproducibly controlled to a high degree of accuracy; and
- 30       (6) for the deposition of an amorphous DLC coating onto the surface of a transducer assembly, which surface faces the magnetic recording media, in which the protective amorphous diamond-like carbon coating is manufactured over large areas with high throughput.

The ion beam deposited DLC coating protects the magnetic transducer assembly from wear and corrosion damage during normal operation and significantly extends the lifetime of the magnetic transducer system. Additionally, the method for manufacture of the ion beam deposited DLC coating substantially

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reduces or eliminates the disadvantages and shortcomings of prior art DLC coating methods. It is not intended by the discussion of a particular transducer assembly to limit the method of the present invention to any particular type of transducers, sliders, or tape heads.

5 It has been unexpectedly found that the ion beam deposition process for the DLC coatings of the present invention produced remarkable performance on a variety of magnetic transducers and magnetic transducer assemblies such as sliders and tape heads. The remarkable performance compared to prior art techniques is the result of the combination of the critical features and attributes listed below.

10 The method of the present invention is capable of:

(1) Overcoming the difficulties in obtaining an atomically clean surface by sputter-etching the substrates using an ion beam of controlled shape, current, and energy. The ion beam "shape" is controlled by focusing the beam with electrostatic or magnetic fields. In this way, the beam can be efficiently delivered  
15 to the substrates to be processed, with maximum utilization. Control of ion beam current and beam energy to within 1% is routinely achieved which results in a highly repeatable and predictable rate of removal of surface residual hydrocarbons and other contaminant layers. In addition, the ion beam sputter-etching process is conducted in high vacuum conditions, such that oxidation or contamination of the  
20 transducer surface with residual gases in the coating system is negligible. Finally, the apparatus geometry can be easily configured such that the sputtered contaminants deposit on the vacuum chamber walls, and do not re-deposit onto the surface of the part as it is being sputter-etched.

(2) Producing excellent adhesion of the protective ion beam deposited DLC  
25 layer by generating an atomically clean surface prior to the deposition of the coating, and via the use of silicon-containing adhesion-promoting interlayers between the DLC coating and the substrate. The silicon-containing layers are preferably deposited by ion beam sputter deposition immediately upon completion of the ion beam sputter-etching step to achieve maximum density and adhesion to  
30 the substrate. Deposition of the adhesion-promoting interlayers immediately upon completion of the ion beam sputter-etching step minimizes the possibility for re-contamination of the sputter-etched surface with vacuum chamber residual gases

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or other contaminants. The silicon-containing layers are selected from the group consisting of amorphous silicon, silicon oxide, silicon nitride, silicon oxy-nitride, silicon carbide, silicon carbonitride, the so-called silicon-doped DLC, mixtures thereof and chemical combinations thereof. Each of the silicon-containing  
5 interlayers may contain hydrogen.

(3) Producing highly dense ion beam deposited DLC coatings. This makes the coatings excellent barriers to water vapor and oxygen. The excellent barrier properties of the thin ion beam deposited DLC coatings presumably result from the extremely high degree of ion bombardment during film growth, compared to prior  
10 art methods. It has been found that DLC coatings prepared by direct ion beam deposition from methane gas in accordance with the present invention have extremely low permeability to water vapor and oxygen. It was demonstrated that 250 Å-thick direct ion beam deposited DLC films having hardness in the range of approximately 10-12 GPa decreased the oxygen permeability of polyethylene and  
15 polypropylene plastic film sheets by greater than 50 times. DLC coatings as thin as 50 Å provided similar results. The plastic sheet substrates for this test were chosen especially for ease of determination of the permeability of the DLC coatings to oxygen and water vapor. In addition to their high density, the ion beam deposited DLC coatings of the present invention are also exceedingly  
20 smooth, which produces a surface with high resistance to wear.

(4) Producing coherent, dense ion beam DLC coatings having thickness of 50 Å or less and providing magnetic transducers with excellent wear protection. This result is also presumably due to the extremely high degree of ion bombardment during film growth, compared to prior art methods. Ultra-low  
25 thickness protective layers are critically important for the newest technology of ultra-high density magnetic recording media, in which the required distance between the magnetically active surface of the magnetic transducer material and the top surface of the recording medium can be as low as 100 Å or less. The newest technology of direct contact tape heads is the most stringent example where this  
30 dimension is minimized.

(5) Producing highly electrically non-conducting DLC coatings as well as adhesion promoting interlayers which preferably are electrically non-conducting.

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Use of these electrically non-conducting interlayers provides improved performance of magnetoresistive sliders, magnetoresistive tape heads, and other transducers, compared to prior art methods.

(6) Producing a coating in which its properties do not change with layer thickness as is found for the prior art RF plasma deposition processes. This attribute is achieved because the coating deposition step is preferably conducted with a charge neutralized ion beam. The use of charge neutralized ion beam deposition process also allows for coating of parts with complex geometry without interference to the process. Parts of varying geometry can be coated within a single coating run with no adverse effect on the deposition conditions. Complete slider assemblies, tape heads, or other transducer assemblies can be easily coated. In addition, on substrates which contain electrically conducting and electrically insulating materials, all portions can be coated with the same high-quality DLC coating. In the case of the plasma deposition methods, DLC coatings of different properties may be deposited on different locations of the same substrate, depending upon whether the area being coated is an electrical conductor or an electrical insulator, and the electrical connections between the substrates and the vacuum chamber. The lack of substrate geometry constraints of the present invention is in sharp contrast to the plasma deposition methods of the prior art.

(7) Having magnetic transducer assemblies fixtured for coating with ease. Because of the ease of fixturing transducer substrates of nearly any shape or configuration, the ion beam process of the present invention can be used to apply a DLC coating to the transducer during any part of the magnetic transducer fabrication or assembly process. For example, the DLC coating can be applied during fabrication of the transducer element as in U.S. Pat. Nos. 5,159,508 and 5,175,658, after fabrication of the transducer element, but before completion of the final transducer assembly, or after completion of the final transducer assembly.

(8) Obtaining minimal batch-to-batch variation in the properties of the DLC coatings. This is the case because process parameters such as ion energy, ion current density, gas flow rate, and deposition chamber pressure are largely decoupled in the ion beam deposition method of the present invention, and because each of these process parameters can be accurately controlled and reproduced to a

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high degree of certainty, often to within 1%. In addition, the process endpoint DLC coating thickness is easily defined and reproduced.

(9) Producing high part-to-part thickness uniformity, e.g. a variation of less than 2% can be easily achieved. This is the case because of the compatibility of the method of the present invention with commercially available substrate holders incorporating motion, i.e. rotation and/or planetary motion.

(10) Being readily scaled-up to accommodate mass production because large scale ion beam sources are commercially available. For example, commercially available 38 cm ion beam sources have been used to deposit DLC coatings simultaneously over four 18-inch diameter platens with a thickness variation across all parts of less than +/- 2%. Similar ion beam sources can be used to practice the process of the present invention. Plasma deposition systems for DLC coatings are not presently commercially available on this scale.

The apparatus for carrying out the preferred embodiment form of the invention is illustrated schematically in FIG.1. The coating process is carried out inside a high vacuum chamber 1 which is fabricated according to techniques known in the art. Vacuum chamber 1 is evacuated into the high vacuum region by first pumping with a rough vacuum pump (not shown) and then by a high vacuum pump 2. Pump 2 can be a diffusion pump, turbomolecular pump, cryogenic pump ("cryopump"), or other high vacuum pumps known in the art. The use of cryopumps with carbon adsorbents is somewhat less advantageous than other high vacuum pumps because those cryopumps have a low capacity for hydrogen which is generated by the ion beam sources used in the present invention for the deposition of DLC. The low capacity for hydrogen results in the need to frequently regenerate the adsorbent in the cryopumps.

It is understood that the process of the present invention can be carried out in a batch-type vacuum deposition system, in which the main vacuum chamber is evacuated and vented to atmosphere after processing each batch of parts; a load-locked deposition system, in which the main vacuum deposition chamber is maintained under vacuum at all times, but batches of parts to be coated are shuttled in and out of the deposition zone through vacuum-to-air load locks; or in-line processing vacuum deposition chambers in which parts are flowed constantly from

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atmosphere, through differential pumping zones, into the deposition chamber, back through differential pumping zones, and returned to atmospheric pressure.

Transducer substrates to be coated are mounted on substrate holder 3, which may incorporate tilt, simple rotation, planetary motion, or combinations thereof.

5 The substrate holder can be in the vertical or horizontal orientation, or at any angle in between. Vertical orientation is preferred to minimize particulate contamination of the substrates, but if special precautions such as low turbulence vacuum pumping and careful chamber maintenance are practiced, the substrates can be mounted in the horizontal position and held in place by gravity. This horizontal  
10 mounting is advantageous from the point of view of easy fixturing of small substrates such as sliders which have just been separated from the chip. This horizontal geometry can be most easily visualized by rotating the illustration in FIG.1 by 90 degrees.

Prior to deposition, the transducer substrates are ion beam sputter-etched  
15 with an energetic ion beam generated in ion beam source 4. Ion beam source 4 can be any ion source known in the prior art, including Kaufman-type direct current discharge ion sources, radio frequency or microwave frequency plasma discharge ion sources, each having one, two, or three grids, or gridless ion sources such as the End Hall ion source of U.S. Pat. No. 4,862,032. The ion source beam  
20 is charge neutralized by introduction of electrons into the beam using a neutralizer (not shown), which may be a thermionic filament, plasma bridge, hollow cathode, or other types known in the prior art.

Ion source 4 is provided with inlets for introduction of inert gases 5, such as argon, krypton, and xenon, for the sputter-etching, and hydrocarbon gases 6,  
25 such as methane, acetylene, and similar gas for the deposition of DLC. During the deposition of DLC, the hydrocarbon gas can be mixed with an inert gas, and/or hydrogen or nitrogen (inlets not shown) to modify the properties of the resultant DLC coating. As in U.S. Pat. No. 4,490,229, an additional ion source (not shown) can be used to co-bombard the substrates during DLC deposition to alter the film  
30 properties.

An additional ion beam source 7, is also provided for ion beam sputter-deposition of interlayer materials onto the substrates. Ion beam source 7 is

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provided with inlets for operation on inert gases 8, such as argon, krypton, and xenon, and for reactive gases 9, such as nitrogen and oxygen. The ion beam from source 7 is directed onto a sputtering target 10, which can be silicon, silicon nitride, or silicon carbide. A silicon target is preferable. Reactive gases such as nitrogen, oxygen, and methane can be introduced into the vacuum chamber background during the reactive sputter deposition of the silicon nitride, silicon carbide, and silicon oxynitride to react with the depositing layer and modify its stoichiometry and properties.

According to the method of the present invention, after completion of the lapping, or other operation to define the magnetic pole pieces, the surface of the magnetic transducer substrate is first chemically cleaned to remove contaminants. Ultrasonic cleaning in solvents, or other detergents as known in the art is often effective; details of the cleaning depend upon the nature of the contamination and residue remaining on the part after lapping and handling. It has been found that it is critical for this step to be effective in removing surface contaminants and residues, or the resulting adhesion of the DLC coating will be poor. This is because in many transducer designs, only a very small amount, e.g.  $<100 \text{ \AA}$ , of surface material can be removed during the subsequent in-vacuum cleaning by ion beam sputter-etching step in order to minimize pole recession.

In the second step, the substrate is inserted into a vacuum chamber, and the air in said chamber is evacuated. Typically, the vacuum chamber is evacuated to a pressure of about  $1 \times 10^{-5}$  Torr or less to ensure removal of water vapor and other contaminants from the vacuum system. However, the required level of vacuum which must be attained prior to initiating the next step must be determined by experimentation. The exact level of vacuum is dependent upon the nature of the substrate material, the sputter-etching rate, the constituents present in the vacuum chamber residual gas, and the details of the adhesion-promoting interlayer.

In the third step, the substrate surface is bombarded with energetic gas ions to assist in the removal of residual contaminants, e.g. any residual hydrocarbons, surface oxides and other contaminants, and to activate the surface. This sputter-etching of the transducer substrate surface is required to achieve high adhesion of interlayer. The sputter-etching can be carried out with inert gases such

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as argon, krypton, and xenon. Additionally, hydrogen may be added to the ion beam to assist in activation of the surface. Typically, in order to achieve efficient and rapid ion sputter-etching, the ion beam energy is greater than 20 eV. Ion energies as high as 2000 eV can be used, but ion beam energies in the range of  
5 about 20 to about 500 eV result in the least amount of atomic scale damage to the transducer substrate.

Once the desired throat height dimension is achieved by the lapping or other process used in fabrication of the transducer, this should not be significantly affected by subsequent processing of the head. Due to the higher ion beam etch  
10 rates of magnetic materials compared to ceramics, a phenomenon known as "pole recession" can occur if the ion beam sputter-etching process is operated at the incorrect condition, or for an extended period of time. Minimizing the sputter-etching time to remove  $<100 \text{ \AA}$  of material, operating with a beam of heavy inert gas (such as xenon) ions, and varying ion beam energy and angle can be used  
15 to minimize the effects of differential etching and pole recession.

Immediately after the transducer substrate surface has been sputter-etched, a silicon-containing interlayer material is deposited. It has been found that deposition of interlayer materials which contain silicon atoms onto the substrate prior to deposition of the DLC layer results in highly adherent DLC coatings with  
20 outstanding wear resistance properties. It is currently believed that reaction between silicon atoms in the interlayer material and the carbon atoms in the DLC layer is essential for the DLC coating to exhibit excellent adhesion.

The thickness of the silicon-containing interlayer can be in the range of about  $10 \text{ \AA}$  to about  $500 \text{ \AA}$  in thickness. However, in order to provide the  
25 minimum spacing between the magnetic transducer and the magnetic recording medium, thinner layers, e.g. about  $10 \text{ \AA}$  to about  $50 \text{ \AA}$ , are preferred.

The silicon-containing interlayer can be deposited by a variety of processes, including magnetron sputtering, plasma deposition, direct ion beam deposition, or ion beam sputter deposition. The silicon-containing interlayer is preferably  
30 deposited by either direct ion beam deposition, or ion beam sputter deposition. Direct ion beam deposition of interlayers containing silicon and one or more of the elements hydrogen, oxygen, carbon, and nitrogen can be performed by operating

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ion source 4 on gases which contain these elements. For example, ion source 4 can be operated on diethylsilane gas to produce an interlayer containing silicon, carbon, and hydrogen. It is believed that the ion beam sputter deposition process produces silicon interlayers or interlayers containing silicon and one or more of the  
5 elements hydrogen, oxygen, carbon, and nitrogen with high density and improved adhesion to the underlying transducer substrate due to the high energy ion bombardment associated with this process.

It has been found that excellent adhesion of the DLC coating to the substrate can be obtained by using electrically non-conducting layers of  
10 silicon-containing materials. Use of these layers is not known in the prior art. These electrically non-conducting silicon-containing interlayers are advantageous for use in magnetoresistive sliders, magnetoresistive tape heads, and other transducers for the reasons discussed above.

Examples of these electrically non-conducting silicon-containing layers  
15 include silicon carbide, silicon nitride, silicon oxide, and silicon oxy-nitride, and mixtures thereof and chemical combinations thereof, such as "silicon carbonitride". By "silicon carbide", it is meant to include materials which are composed of the elements silicon and carbon, and possibly hydrogen. Stoichiometric and non-stoichiometric amounts of silicon and carbon are included in the definition of  
20 this silicon carbide material. By "silicon nitride", it is meant to include materials which are composed of the elements silicon and nitrogen, and possibly hydrogen. Stoichiometric and non-stoichiometric amounts of silicon and nitrogen are included in the definition of this silicon nitride material. By "silicon oxide", it is meant to include materials which are composed of the elements silicon and oxygen, and  
25 possibly hydrogen. The definition of silicon oxide includes only materials which have a higher atomic concentration of silicon than does silicon dioxide,  $\text{SiO}_2$ . It was found that attempts to use a sputtered silicon dioxide interlayer produced a DLC coating with poor adhesion. It is believed that a silicon oxide material having excess silicon atoms which are not fully bonded to oxygen is required to produce a  
30 DLC coating on magnetic transducers with optimum adhesion. By "silicon oxy-nitride", it is meant to include materials which are composed of the elements silicon, oxygen, and nitrogen, and possibly hydrogen. Materials falling under the

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chemical formula  $\text{SiO}_x\text{N}_y\text{H}_z$  are considered to be within the definition of this silicon oxy-nitride material.

Following completion of the deposition of the silicon-containing interlayer to the desired thickness, a diamond-like carbon top layer is deposited by ion beam deposition. It is important to minimize the time between completion of the silicon-containing interlayer, and the start of the deposition of the DLC layer. Deposition of the DLC layer immediately after completion of the interlayer deposition step minimizes the possibility for re-contamination of the interlayer surface with vacuum chamber residual gases or other contaminants. The thickness of the protective ion beam deposited DLC coating is constrained to small dimensions since the coating thickness adds directly to the spacing between the magnetic transducer and the magnetic recording medium. Depending on the design and operation of the transducer, the DLC coating thickness is typically in the range of about 25 Å to about 2,000 Å. Thicker DLC layers are generally preferable in terms of providing increased protection against wear and corrosion, although outstanding wear and corrosion resistance is also obtained by ion beam deposited DLC coatings at the low end of this thickness range. The actual thickness of the ion beam deposited DLC layer is chosen in practice based on the maximum allowable increase in spacing between the magnetic transducer and the magnetic recording medium.

Several ion beam deposition methods may be used for the formation of the DLC coatings of the present invention, including direct ion beam deposition, direct ion beam deposition with ion assist, i.e. "direct dual ion beam deposition", ion beam sputter deposition from a carbon target, and ion beam sputter deposition with ion assist, i.e. "dual ion beam sputter deposition". The ion beam sputter deposition methods offer excellent control, uniformity, and flexibility of substrate geometry, but the deposition rate is slower than that of the direct ion beam deposition process. To maximize the density and electrical resistivity of the ion beam sputter-deposited DLC coating, bombardment of the growing film by an additional ion beam ("ion assist") is normally required. Filtered carbon cathodic arc ion sources and laser ablation (from a solid carbon target) ion sources can also be used as ion sources for generation of the carbon deposition flux for the DLC coatings of

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the method of the present invention. In both of these ion sources, it is essential to perform some filtering of the beam to remove particles which degrade the coating. These ion sources produce extremely hard diamond-like carbon coatings with very low hydrogen content. However, the coating stress level is high, so extreme care  
5 in substrate chemical cleaning, ion beam pre-cleaning, and interlayer deposition must be taken to obtain excellent coating adhesion.

For sake of process simplicity, rapid deposition, and ease of scale-up to mass production, direct ion beam deposition from a hydrocarbon gas source is the most preferred deposition process for this invention. Methane as the hydrocarbon  
10 source gas is preferred, but other hydrocarbon gases, such as acetylene, butane, and benzene can be used as well. Inert gases and hydrogen may be introduced into the ion source plasma to modify the DLC film properties. The ion energy used in the DLC deposition process may be in the range of approximately 20 eV to approximately 1000 eV. Ion energies in the range of about 20 eV to about 300 eV  
15 are most preferred to minimize heating of the substrate during deposition. Excellent wear resistance and low permeability have been demonstrated by DLC coatings having hardness in the range of about 10 to about 15 GPa using direct ion beam deposition. This is in contrast to prior art references, e.g. U.S. Pat. No. 4,717,622, which require hardness in excess of approximately 20 GPa (2,000  
20 kg/mm<sup>2</sup>) to achieve high wear resistance. In addition to the ion beam for direct deposition, an ion assist beam, as in U.S. Pat. No. 4,490,229, can be utilized but is not required.

Once the chosen thickness of the DLC layer has been achieved, the deposition process on the transducer substrates is terminated, the vacuum chamber  
25 pressure is increased to atmospheric pressure, and the coated magnetic transducer substrates are removed from the vacuum chamber.

#### Examples

The examples and discussion which follow further illustrate the superior performance of the coated products of the method of this invention. The examples  
30 are for illustrative purposes and are not meant to limit the scope of the claims in any way.

#### Example 1

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An  $\text{Al}_2\text{O}_3/\text{TiC}$  slider (IBM 3380-type) was coated with ion beam deposited DLC by the following method. The sliders already mounted onto their suspension system were first chemically cleaned with isopropanol and blown dry with nitrogen. The cleaned sliders were then attached to a 6-inch diameter graphite plate using adhesive tape. The graphite plate was then mounted onto a rotary stage, and the vacuum chamber was evacuated to a pressure of  $4.8 \times 10^{-6}$  Torr. The slider was then sputter-etched using an argon ion beam from an 11 cm Kaufman-type ion source at an energy of 500 eV with a beam current of 137 mA for 2 minutes. The etch rate of a silicon witness coupon under these conditions was approximately 300 Å/minute. After this sputter-etching step, a 1000 eV, 100 mA argon ion beam was used to sputter-deposit a 25 Å thick layer of silicon by ion beam sputter deposition from a silicon target. After deposition of the silicon layer, a 100 Å thick DLC layer was deposited by direct ion beam deposition using the 11 cm Kaufman-type ion source, operated at a beam energy of 75 eV and a beam current of 175 mA.

#### Example 2

An  $\text{Al}_2\text{O}_3/\text{TiC}$  slider (IBM 3380-type) was coated with ion beam deposited DLC by the same procedure as in Example 1, except the vacuum chamber was initially was evacuated to a pressure of  $5.0 \times 10^{-6}$  Torr, and the thickness of the DLC coating was 50 Å.

#### Example 3

A magnetoresistive tape head was chemically cleaned, and mounted in an aluminum fixture. The fixture was installed in a stainless steel vacuum chamber and the chamber was evacuated to a pressure of  $3.0 \times 10^{-6}$  Torr. The tape head was then sputter-etched using an argon ion beam from an 11 cm Kaufman-type ion source at an energy of 500 eV with a beam current of 137 mA for 15 seconds. The etch rate of a silicon witness coupon under these conditions was approximately 300 Å/minute. After this sputter-etching step, a 1500 eV, 50 mA nitrogen ion beam was used to sputter-deposit a 25 Å thick layer of silicon nitride by reactive ion beam sputter deposition from a silicon target. After deposition of the silicon nitride layer, a 225 Å thick DLC layer was deposited by direct ion beam deposition

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using the 11 cm Kaufman-type ion source, operated at a beam energy of 75 eV and a beam current of 50 mA.

#### Example 4

Analog tape heads were cleaned with isopropanol and then blown dry with  
5 nitrogen gas. The samples were mounted in an aluminum fixture and the fixture was installed into a stainless steel vacuum chamber which was evacuated to a pressure of  $4.6 \times 10^{-6}$  Torr. The tape heads were then sputter-etched for one minute using a 500 eV, 137 mA argon ion beam generated by an 11 cm Kaufman type ion source. The etch rate of a silicon witness coupon under these conditions  
10 was approximately 300 Å/minute. After this sputter-etching step, a 1000 eV, 100 mA argon ion beam was used to sputter-deposit a 200 Å thick layer of amorphous silicon by ion beam sputter deposition from a silicon target. After deposition of the silicon layer, a 1200 Å thick DLC layer was deposited onto the tape heads by direct ion beam deposition using the 11 cm Kaufman-type ion source, operated at a  
15 beam energy of 75 eV and a beam current of 100 mA.

The durability of the ion beam deposited DLC coating of the present invention was demonstrated by applying the coating to  $\text{Al}_2\text{O}_3/\text{TiC}$  sliders used as transducers with magnetic recording disks. The outstanding durability and performance enhancements due to the DLC coating of the present invention were  
20 determined by performing two types of accelerated wear tests: a constant speed drag test, and a contact-start-stop (CSS) test. The CSS test is used by the magnetic disk recording industry as the ANSI standard method. A typical mode of failure in the tests is build-up of friction with revolutions of drag or cycles of CSS. For uncoated sliders, the friction starts at a coefficient level of approximately 0.2 and  
25 builds up to three or four times that initial value with several thousand cycles. The high levels of friction cause particle pullout with resulting abrasive wear and failure of the head-disk interface. Even if such wear does not occur, the high levels of friction are unacceptable, because the drive motor may not have sufficient torque to start the drive from the rest condition. Therefore, it is desirable to find a  
30 combination of protective overcoat and lubricant to achieve a high level of CSS or drag revolutions without unacceptable friction build-up.

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Uncoated and DLC-coated sliders were measured on a standard tester developed by Hewlett Packard Laboratories, which consists of a computer-controlled spindle on which the disk is clamped and the slider-suspension assembly is attached to a strain gauge instrumented load beam to measure normal and frictional forces on the slider. Tests were conducted in a class 100 clean hood in room air at 28°C and 35% relative humidity. Standard commercially available 95 mm diameter thin film disks with a 200-300 Å thick layer amorphous carbon and a 20 Å thick layer of lubricant were used in the tests.

The test procedure used was as follows. After the disk had been clamped to the spindle and the slider is loaded on the disk, a single revolution is made at low rpm during which the friction force of the dragging slider is recorded. Then a single CSS is made and the friction is measured during the first revolution to reveal the peak static friction, called the "stiction". An additional indicator of failure is the touch-down velocity (TDV), defined as the speed at which friction reaches a threshold value upon landing of the slider as the spindle revolution speed is reduced from the normal operating speed.

Next, the tester was programmed to perform either slow speed drag tests or CSS tests while taking one-revolution friction data periodically, at 100 cycle intervals. These data are averaged, and the result is plotted as a function of revolutions along with the TDV. An example of drag test data for an uncoated IBM 3380-type  $\text{Al}_2\text{O}_3$ -TiC slider is presented in FIG. 2A, and 2B, and an example of CSS test data for an uncoated IBM 3380-type  $\text{Al}_2\text{O}_3$ -TiC slider is presented in FIG. 2C and 2D.

Note in FIG. 2A that the frictional force started at 2.07 gmf and increased with the number of test revolutions to a value near 5 gmf at 800 revolutions. Then, it remained nearly constant until increasing to a maximum of 6 gmf at approximately 2600 revolutions. The test was stopped at 3300 revolutions when the TDV showed two consecutive values 50% above the lowest value, a criterion chosen to maximize the accuracy of the determination of contamination on the sliders by wear products. An increase of TDV is an indication that material has transferred from the disk to the slider. Similarly, in FIG. 2C, the frictional force started at 1.93 gmf and increased to 5.2 gmf at approximately 300 start/stops. It

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reached a value near 7 gmf before the test was stopped after the TDV reached the termination criteria 1200 start/stops. Microscopic examination of the sliders after these tests revealed that there was a significant amount of wear debris adhering to the rails of the sliders.

5 IBM 3380-type  $\text{Al}_2\text{O}_3$ -TiC sliders coated in Example 1 (100 Å layer of DLC) and Example 2 (50 Å layer of DLC) by the ion beam deposition process of the present invention were evaluated using the same test conditions used for the uncoated sliders. The tests on the DLC-coated sliders were set to terminate at 30,000 test revolutions if not interrupted by the increase of the TDV. FIG. 3A and  
10 FIG. 3B show the drag test results for a slider coated with 100 Å of DLC in Example 1. The test result is substantially different and improved relative to the uncoated sliders. In FIG. 3A, the frictional force rapidly increased from 1.61 gmf to about 3.2 gmf and remained essentially constant for 30,000 revolutions. In FIG. 3B, the TDV decreased from 3.51 meters/second to 2.8 m/s and remained  
15 essentially constant for the duration of the test. After 30,000 cycles was completed, only a small amount of wear material was found on one rail of the slider. The rails were much cleaner than was the case for the tests of the uncoated sliders.

FIG. 4A and FIG. 4B show the drag test results for a slider coated with 50  
20 Å of DLC in Example 2. The test results were nearly identical to those found in FIG. 3A and FIG. 3B for the slider coated with 100 Å of DLC in Example 1. Again, there was only a very small amount of debris found on the rails of the slider for this test. The rails were much cleaner than was the case for the tests of the uncoated sliders.

25 FIG. 2C and FIG. 2D show the results of a CSS test for an uncoated  $\text{TiC}/\text{Al}_2\text{O}_3$  slider. It was found that the frictional force increased from an initial value of 1.93 gmf to 5.4 gmf at approximately 400 CSS cycles, then increased more gradually until about 800 cycles, after which it became erratic and the test was terminated by the TDV criterion at 1,200 CSS cycles. After termination of  
30 this CSS test, there was much more debris found on the rails of this slider than was found in the case of the previous drag tests on uncoated sliders.

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FIG. 5A and FIG. 5B show the results of CSS tests on the same slider (100 Å DLC coating) used to obtain the data in FIG. 3A and FIG. 3B. The data show that the frictional force increased rapidly from 1.77 gmf to approximately 3.3 gmf and remained essentially constant for 8,000 CSS cycles. The TDV showed only one instance of an increase, but otherwise showed a gradual decrease over the duration of the test.

These test results showed a remarkable difference in performance of the ion beam DLC-coated sliders of the present invention compared to uncoated sliders in both drag tests and CSS tests. Other coated and uncoated sliders were also tested with similar results. In the drag tests, all of the uncoated sliders showed an increase of the friction to 6 gmf or more within the first 3,000 revolutions. This represented a 3-fold increase from the initial friction value. On the other hand, the tests with the DLC-coated sliders of the present invention showed a rapid increase to slightly above 3 gmf, but the friction then remained constant for 30,000 revolutions. An increase to 6 gmf would be unacceptable even if the TDV failure criterion had not been met. However, an increase to 3 gmf is tolerable, and the results indicate that the tests could have progressed to significantly higher number of revolutions before a further increase in friction would occur. If doubling of initial friction is set as the failure criterion in the drag tests, the uncoated sliders all failed by 1,500 revolutions, while the ion beam DLC-coated sliders did not fail by 30,000 revolutions. Therefore, a factor of improvement of greater than 20 for the ion beam DLC-coated sliders was indicated by the drag tests.

Similarly, in the CSS tests, the uncoated sliders showed an increase of frictional force to 6 gmf by 1,000 cycles for three tests which were conducted. For the case of the ion beam DLC-coated slider, the frictional force did not increase above 3.4 gmf at any time up to the point the test was terminated at 8,000 cycles. Again, if doubling of the initial frictional force from 2 gmf to 4 gmf is set as the failure criterion, all of the uncoated sliders failed by 200 CSS cycles, whereas the ion beam DLC-coated slider did not yet fail by 8,000 cycles. Therefore, a factor of improvement of greater than 40 for the ion beam DLC-coated sliders was indicated by the CSS tests.

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From the foregoing description, one of ordinary skill in the art can easily ascertain that the present invention provides an improved method for producing highly protective and wear resistant DLC coatings on magnetic transducers. Highly important technical advantages of the method of the present invention include

5 outstanding adhesion of the ion beam deposited DLC coating, and ease and flexibility of mass production of DLC-coated magnetic transducers.

Without departing from the spirit and scope of this invention, one of ordinary skill in the art can make various changes and modifications to the invention to adapt it to various usages and conditions. As such, these changes and

10 modifications are properly, equitably, and intended to be, within the full range of equivalents of the following claims.

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## WHAT IS CLAIMED IS:

1. A method for producing a protective, wear resistant diamond-like carbon coating on the wear surface of a magnetic transducer comprising the steps of chemically cleaning the surface of said magnetic transducer to remove  
5 contaminants, sputter-etching the surface of said magnetic transducer in an evacuated deposition vacuum chamber with a beam of energetic gas ions to further remove residual contaminants, depositing a silicon-containing adhesion-promoting interlayer material, ion beam depositing an electrically non-conducting  
10 diamond-like carbon outer layer to a predetermined thickness, increasing the vacuum chamber pressure to atmospheric pressure and recovering a diamond-like carbon coated magnetic transducer having improved resistance to wear, abrasion and corrosion.
2. The method of Claim 1 wherein said contaminants are selected from the group consisting of residual hydrocarbons, surface oxides and other unwanted  
15 materials.
3. The method of Claim 1 wherein said adhesion-promoting interlayer is deposited by ion beam sputter deposition.
4. The method of Claim 1 wherein said interlayer is deposited by direct ion beam deposition.
- 20 5. The method of Claim 1 wherein said interlayer consists of amorphous silicon.
6. The method of Claim 1 wherein said interlayer is selected from the group consisting of amorphous silicon carbide, silicon nitride, silicon oxide, silicon oxy-nitride, mixtures thereof and chemical combinations thereof.
- 25 7. The method of Claim 1 wherein said outer layer is deposited by direct ion beam deposition from a hydrocarbon gas.
8. The method of Claim 1 wherein said outer layer is deposited by direct ion beam deposition using ions generated by a filtered cathodic arc carbon ion source.
- 30 9. The method of Claim 1 wherein said outer layer is deposited by direct ion beam deposition using ions generated by a laser ablation carbon ion source.

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10. The method of Claim 1 wherein said outer layer is deposited by ion beam sputter deposition from a carbon sputtering target.
11. The product manufactured by the method of Claim 1.
12. The product of Claim 11, wherein said magnetic transducer consists of  
5 a slider.
13. The product of Claim 11 wherein said magnetic transducer consists of a tape head.
14. The product of Claim 11, wherein said magnetic transducer consists of a magnetoresistive slider.
- 10 15. The product of Claim 11 wherein said magnetic transducer consists of a magnetoresistive tape head.
16. A method for producing a protective, wear resistant diamond-like carbon coating on the wear surface of a magnetic transducer comprising the steps of:
- 15 (a) chemically cleaning the surface of said magnetic transducer to remove contaminants;
- (b) mounting said magnetic transducer in a deposition vacuum chamber and evacuating the air from said chamber;
- (c) sputter-etching the surface of said magnetic transducer to further remove  
20 residual contaminants;
- (d) depositing an electrically non-conducting adhesion-promoting interlayer;
- (e) depositing an electrically non-conducting diamond-like carbon outer layer to a predetermined thickness;
- (f) increasing the vacuum chamber pressure to atmospheric pressure; and  
25 (g) recovering a diamond-like carbon coated magnetic transducer having improved wear and corrosion resistance.
17. The method of Claim 16 wherein said interlayer contains silicon.
18. The method of Claim 17 wherein said interlayer is selected from the group consisting of amorphous silicon carbide, silicon nitride, silicon oxide, silicon  
30 oxy- nitride, mixtures thereof and chemical combinations thereof.
19. The method of Claim 16 wherein said sputter-etching process is carried out using an energetic ion beam.

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20. The method of Claim 16 wherein said electrically non-conducting adhesion promoting interlayer material is deposited by plasma deposition.

21. The method of Claim 16 wherein said interlayer is deposited by magnetron sputter deposition.

5 22. The method of Claim 16 wherein said interlayer is deposited by ion beam sputter deposition.

23. The method of Claim 16 wherein said interlayer is deposited by direct ion beam deposition.

10 24. The method of Claim 16 wherein said outer is deposited by direct ion beam deposition from a hydrocarbon gas.

25. The method of Claim 16 wherein said outer layer is deposited by direct ion beam deposition using ions generated by a cathodic arc carbon ion source.

26. The method of Claim 16 wherein said outer layer is deposited by direct ion beam deposition using ions generated from a laser ablation carbon ion source.

15 27. The method of Claim 16 wherein said outer layer is deposited by ion beam sputter deposition from a carbon sputtering target.

28. The method of Claim 16 wherein outer layer is deposited by radio frequency plasma deposition.

20 29. The method of Claim 16 wherein outer layer is deposited by magnetron sputter deposition using a carbon target.

30. The product of the method of Claim 16.

31. The product of the method of Claim 16 wherein said magnetic transducer consists of a magnetoresistive slider.

25 32. The product of the method of Claim 16 wherein said magnetic transducer consists of a magnetoresistive tape head.

33. A transducer for use with magnetic recording media composed of a transducer assembly substrate, an electrically non-conducting adhesion-promoting interlayer deposited on and bonded to said substrate, and a non-conducting diamond-like carbon outer layer deposited on and bonded to said interlayer,  
30 whereby said transducer has improved wear and corrosion resistance.

34. The transducer of Claim 33 wherein said interlayer thickness is in the range of about 10 Å to about 500 Å.

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35. The transducer of Claim 33 wherein said interlayer thickness is in the range of about 10 Å to about 50 Å.

36. The transducer of Claim 33 wherein said outer layer thickness is in the range of about 25 Å to about 2000 Å.

5 37. The transducer of Claim 33 wherein said interlayer contains silicon.

38. The transducer of Claim 37 wherein said interlayer is selected from the group consisting of amorphous silicon carbide, silicon nitride, silicon oxide, silicon oxy-nitride, mixtures thereof and chemical combinations thereof.

10 39. The transducer of Claim 33 wherein said interlayer is deposited by plasma deposition.

40. The transducer of Claim 33 wherein said interlayer is deposited by magnetron sputter deposition.

41. The transducer of Claim 33 wherein said interlayer is deposited by ion beam sputter deposition.

15 42. The transducer of Claim 33 wherein said interlayer is deposited by direct ion beam deposition.

43. The transducer of Claim 33 wherein said outer layer is deposited by direct ion beam deposition from a hydrocarbon gas.

20 44. The transducer of Claim 33 wherein said outer layer is deposited by direct ion beam deposition using ions generated by a cathodic arc carbon ion source.

45. The transducer of Claim 33 wherein said outer layer is deposited by direct ion beam deposition using ions generated from a laser ablation carbon ion source.

25 46. The transducer of Claim 33 wherein said outer layer is deposited by ion beam sputter deposition from a carbon sputtering target.

47. The transducer of Claim 33 wherein said outer layer is deposited by radio frequency plasma deposition.

30 48. The transducer of Claim 33 wherein said outer layer is deposited by magnetron sputter deposition using a carbon target.

49. The transducer of Claim 33 consisting of a magnetoresistive slider.

50. The transducer of Claim 33 consisting of a magnetoresistive tape head.

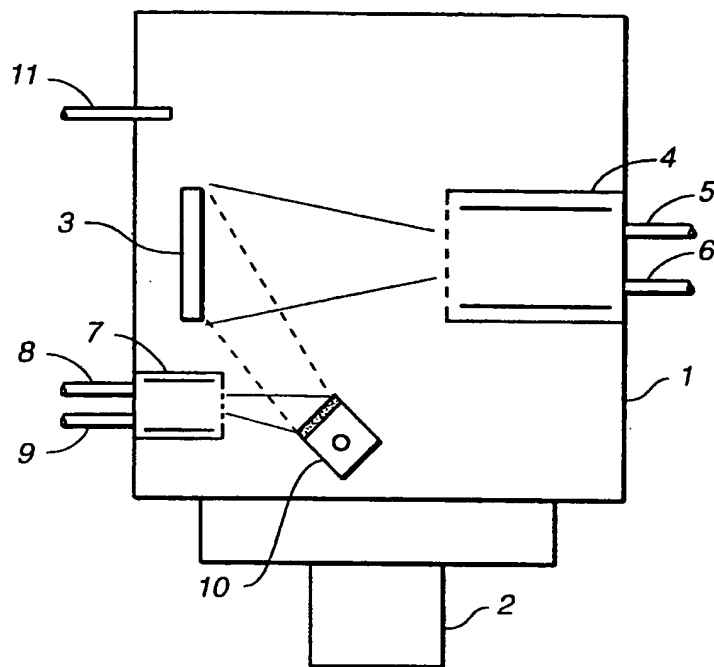
## AMENDED CLAIMS

[received by the International Bureau on 7 August 1995 (07.08.95);  
original claims 1-10, 17-23, 28-32, 39-41 and 46-48 cancelled;  
remaining claims unchanged and renumbered 1 to 22 (2 pages)]

1. A method for producing a protective, wear resistant diamond-like carbon coating on the wear surface of a magnetic transducer comprising the steps of:
  - (a) chemically cleaning the surface of said magnetic transducer to remove  
5 contaminants;
  - (b) mounting said magnetic transducer in a deposition vacuum chamber and evacuating the air from said chamber;
  - (c) ion beam sputter-etching the surface of said magnetic transducer to further remove residual contaminants;
  - 10 (d) ion beam sputter depositing an amorphous, electrically non-conducting adhesion-promoting interlayer selected from the group consisting of silicon, silicon carbide, silicon nitride, silicon oxide, silicon oxy-nitride, mixtures thereof and chemical combinations thereof;
  - (e) direct ion beam depositing an electrically non-conducting diamond-like  
15 carbon outer layer;
  - (f) increasing the vacuum chamber pressure to atmospheric pressure; and
  - (g) recovering a diamond-like carbon coated magnetic transducer having improved wear and corrosion resistance.
2. The method of Claim 1 wherein said outer layer is deposited by direct ion  
20 beam deposition from a hydrocarbon gas.
3. The method of Claim 1 wherein said outer layer is deposited by direct ion beam deposition using ions generated by a cathodic arc carbon ion source.
4. The method of Claim 1 wherein said outer layer is deposited by direct ion beam deposition using ions generated from a laser ablation carbon ion source.
- 25 5. The method of Claim 1 wherein said outer layer is deposited by ion beam sputter deposition from a carbon sputtering target.
6. The product manufactured by the method of Claim 1.
7. The product of Claim 6, wherein said magnetic transducer consists of a slider.
- 30 8. The product of Claim 6 wherein said magnetic transducer consists of a tape head.

9. The product of Claim 6, wherein said magnetic transducer consists of a magnetoresistive slider.
10. The product of Claim 6 wherein said magnetic transducer consists of a magnetoresistive tape head.
- 5 11. A transducer for use with magnetic recording media composed of a transducer assembly substrate, an electrically non-conducting adhesion-promoting interlayer deposited on and bonded to said substrate, and a non-conducting diamond-like carbon outer layer deposited on and bonded to said interlayer, whereby said transducer has improved wear and corrosion resistance.
- 10 12. The transducer of Claim 11 wherein said interlayer thickness is in the range of about 10 Å to about 500 Å.
13. The transducer of Claim 11 wherein said interlayer thickness is in the range of about 10 Å to about 50 Å.
14. The transducer of Claim 11 wherein said outer layer thickness is in the  
15 range of about 25 Å to about 2000 Å.
15. The transducer of Claim 11 wherein said interlayer contains silicon.
16. The transducer of Claim 15 wherein said interlayer is selected from the group consisting of amorphous silicon carbide, silicon nitride, silicon oxide, silicon oxy-nitride, mixtures thereof and chemical combinations thereof.
- 20 17. The transducer of Claim 11 wherein said interlayer is deposited by direct ion beam deposition.
18. The transducer of Claim 11 wherein said outer layer is deposited by direct ion beam deposition from a hydrocarbon gas.
19. The transducer of Claim 11 wherein said outer layer is deposited by direct  
25 ion beam deposition using ions generated by a cathodic arc carbon ion source.
20. The transducer of Claim 11 wherein said outer layer is deposited by direct ion beam deposition using ions generated from a laser ablation carbon ion source.
21. The transducer of Claim 11 consisting of a magnetoresistive slider.
22. The transducer of Claim 11 consisting of a magnetoresistive tape head.

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**FIG. 1**

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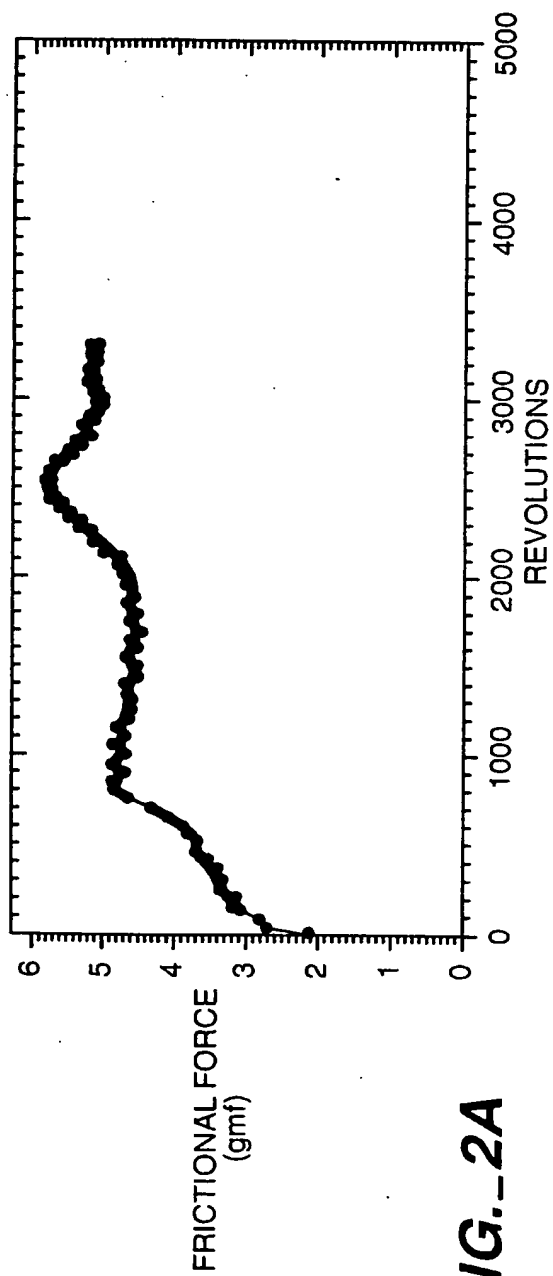


FIG. 2A

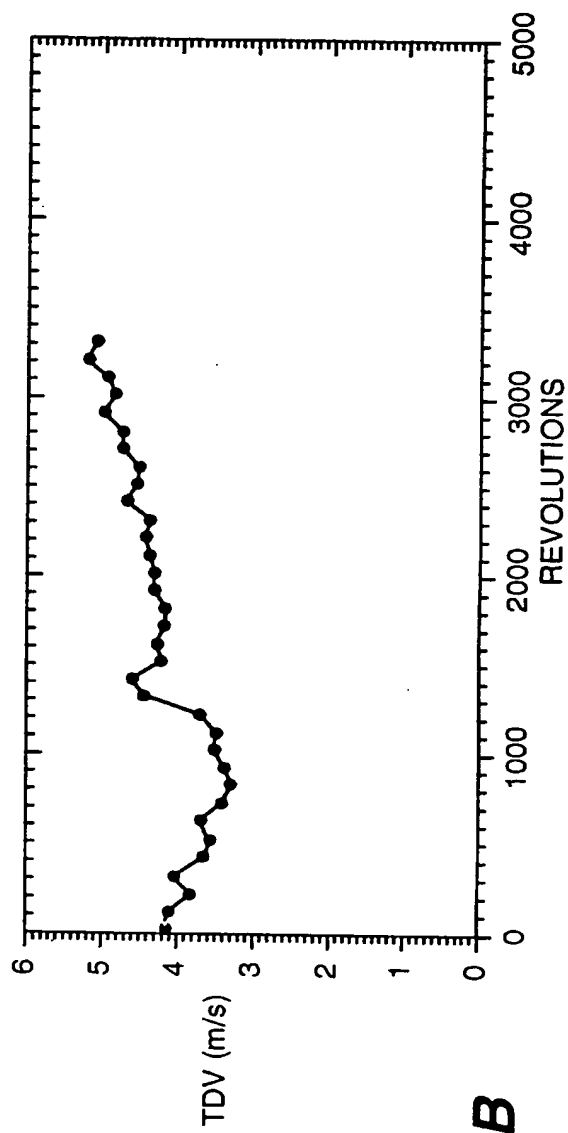


FIG. 2B

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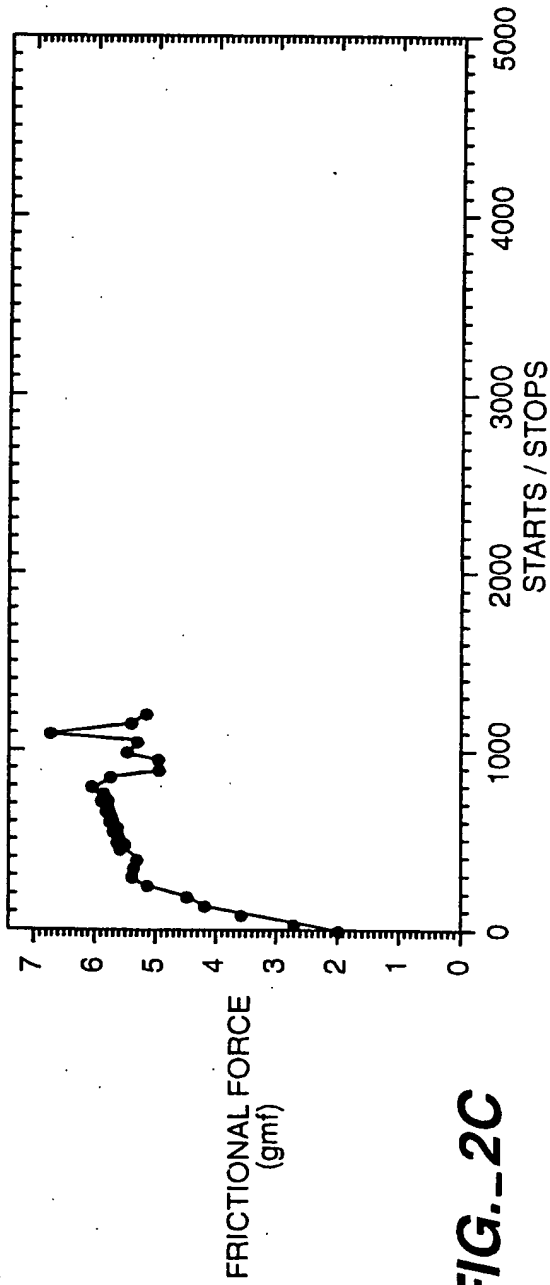


FIG. 2C

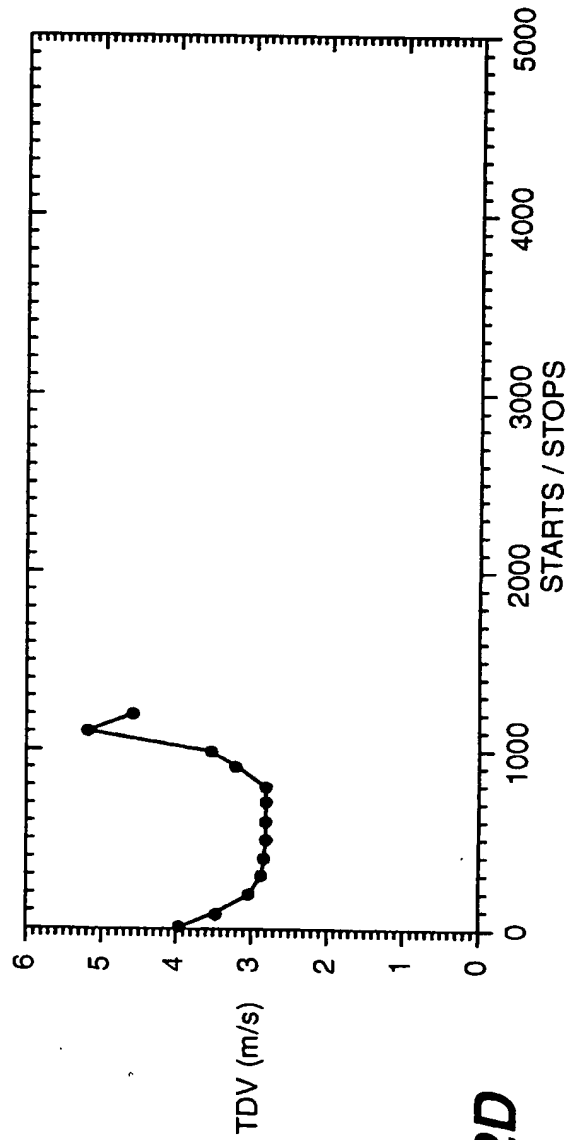
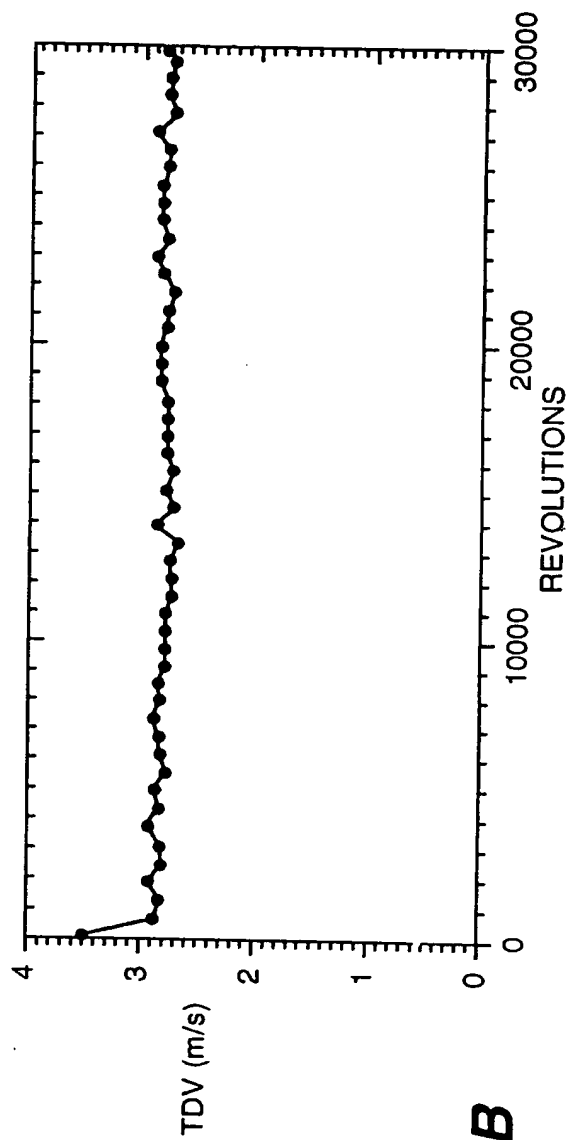
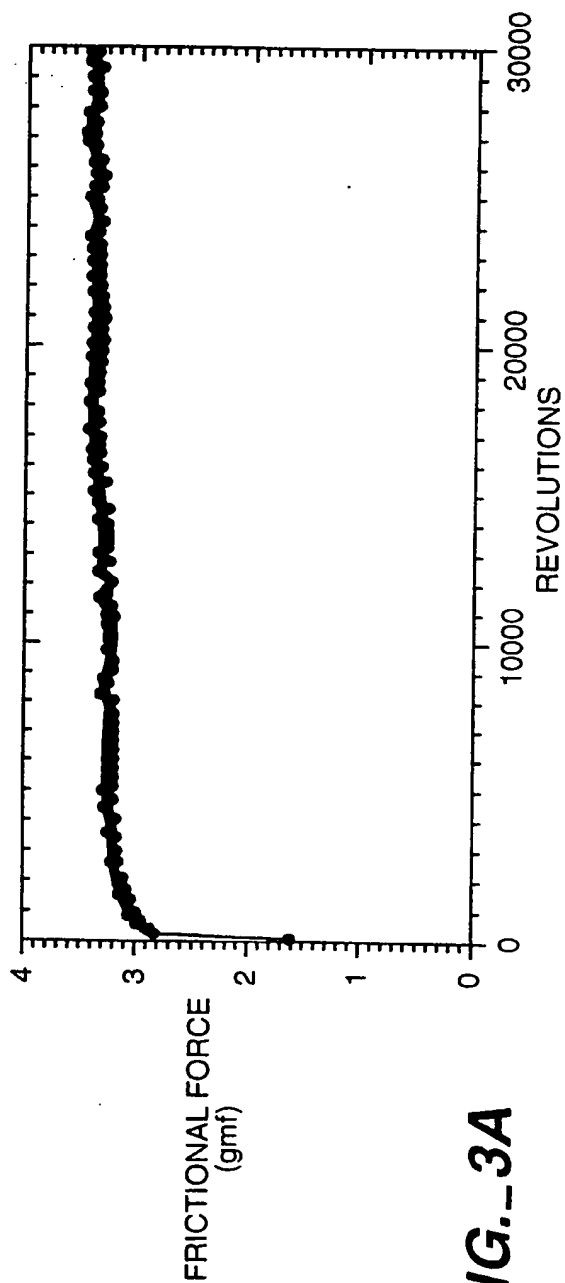
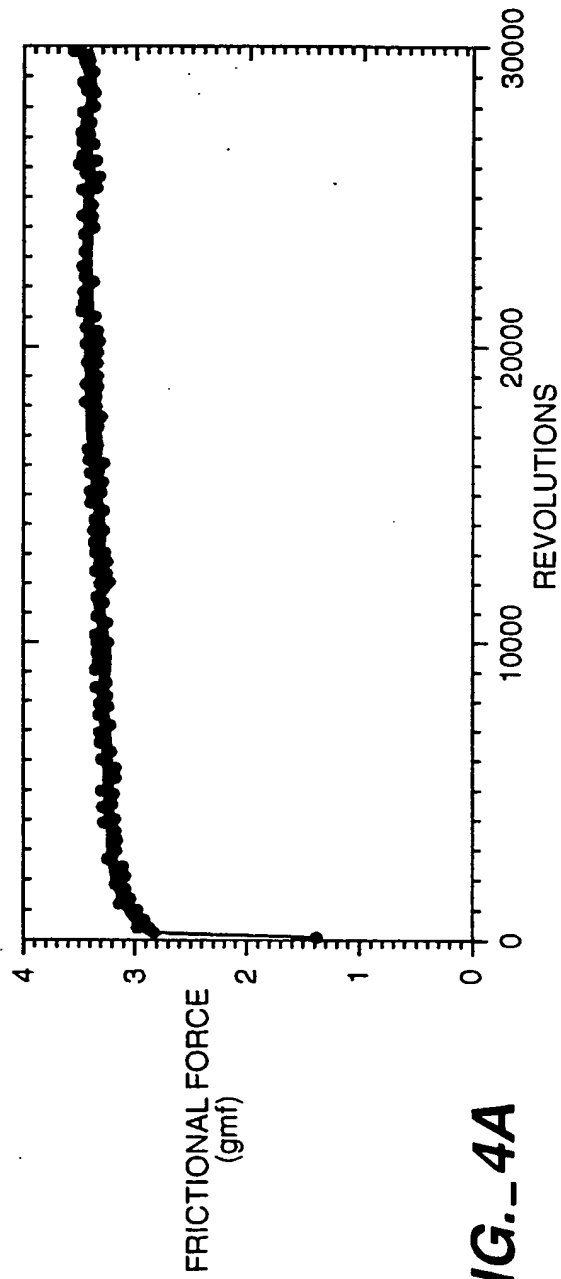
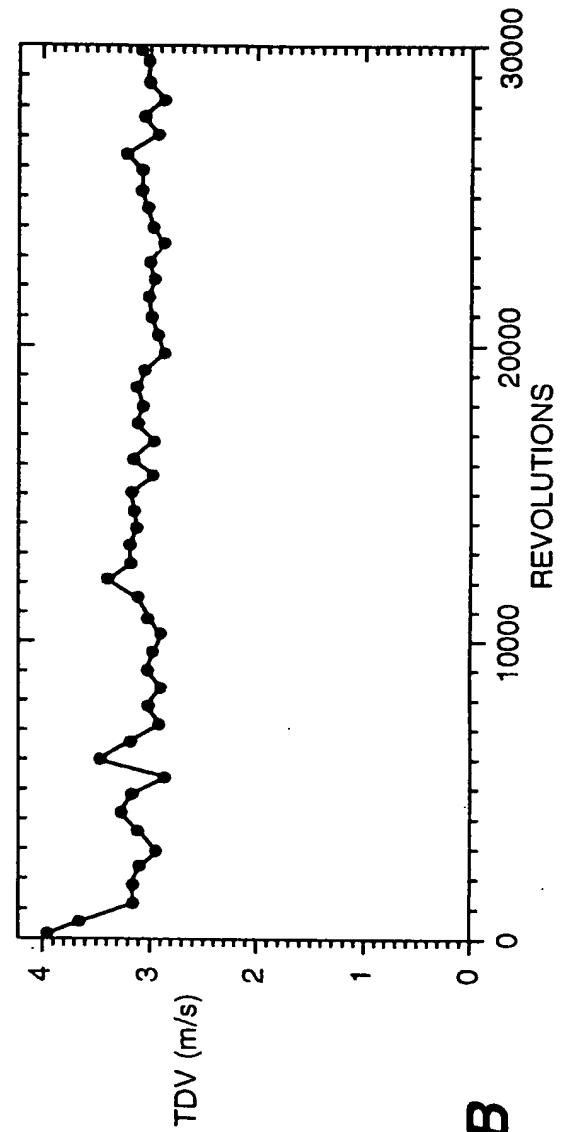


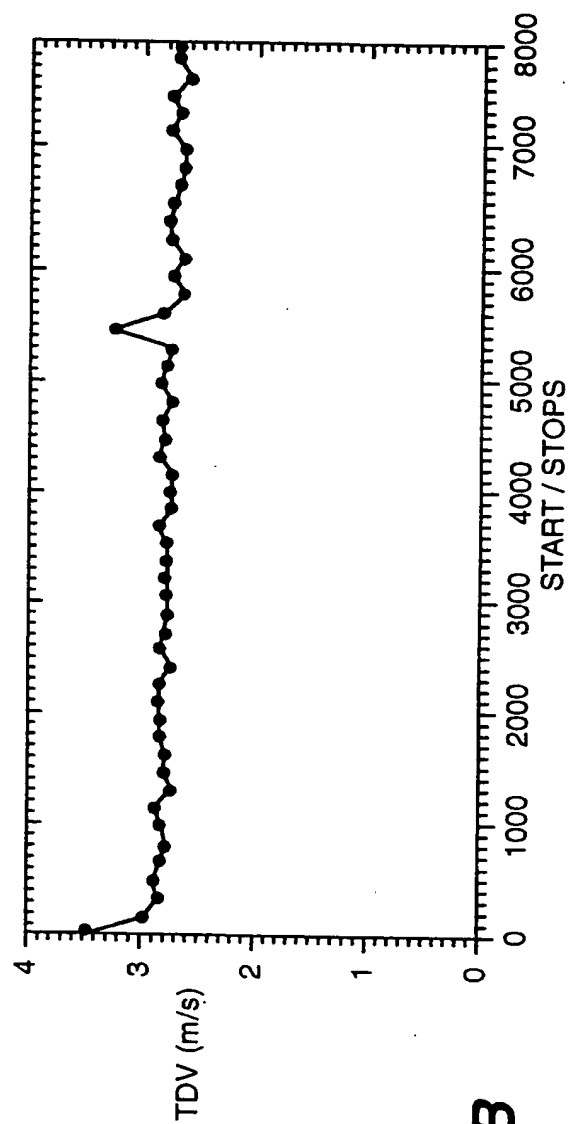
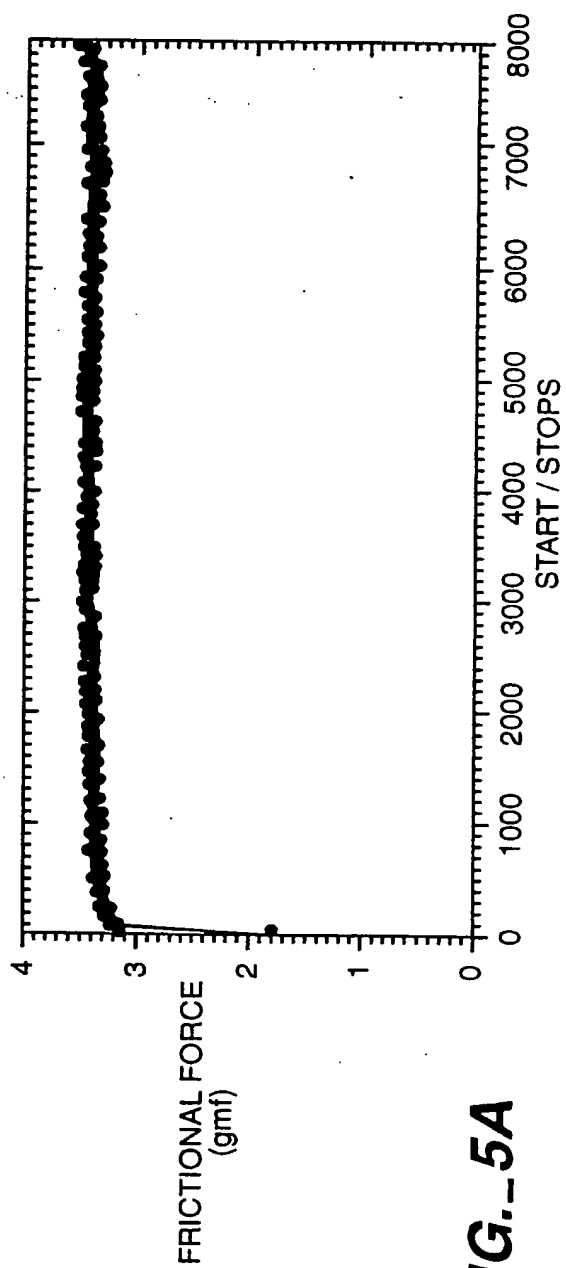
FIG. 2D



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**FIG. 4A****FIG. 4B**

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## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US95/02760

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(6) : C23C 14/00; G11B 5/255

US CL : Please See Extra Sheet.

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : Please See Extra Sheet.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Automated Patent System

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	US, A, 5,336,550 (GANAPATHI ET AL.) 09 AUGUST 1994. (09-08-94) FIGURE 6, COLUMN 9 LINES 1-15.	33-37, 49-50
X ---- Y	US, A, 5,159,508 (GRILL ET AL.) 27 OCTOBER 1992. (10-10-92) FIGURES 5-6, COLUMN 3 LINES 14-18, COLUMN 5 LINES 21-29, COLUMN 5 LINES 35-36, COLUMN 5 LINES 44-51.	33-37, 49-50 ----- 1-7, 9-24, 26-32, 39-43, 45-48
Y	US, A, 5,175,658 (CHANG ET AL.) 29 DECEMBER 1992 (29.12.92) FIGURES 5-6, COLUMN 4 LINES 59-66, COLUMN 5 LINES 38-43	1-7, 9-24, 26-32, 39-43, 45-48

☒ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier document published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&" document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

21 APRIL 1995

Date of mailing of the international search report

19 JUN 1995

 Name and mailing address of the ISA/US  
 Commissioner of Patents and Trademarks  
 Box PCT  
 Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

ROBERT BRUCE BRENNEMAN

Telephone No. (703) 308-3324

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US95/02760

## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 5,135,808 (KIMOCK ET AL.) 04 AUGUST 1992 (04-08-92) COLUMN 1 LINES 31-34, COLUMN 1 LINES 37-40, COLUMN 2 LINES 52-60, COLUMN 3 LINES 32-48, COLUMN 4 LINES 3-21.	1-7, 9-10, 16-24, 26-29, 38
Y	US, A, 4,565,618 (BANKS) 21 JANUARY 1986 (1-21-86) COLUMN 3 LINES 32-40; COLUMN 4 LINES 37-45.	8, 25, 44

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US95/02760

## A. CLASSIFICATION OF SUBJECT MATTER:

US CL :

204/192.11, 192.12, 192.15, 192.16, 192.38; 427/577, 586, 360/102, 103,

## B. FIELDS SEARCHED

Minimum documentation searched

Classification System: U.S.

204/192.1, 192.11, 192.12, 192.15, 192.16, 192.2, 192.23, 192.3, 192.38; 427/523, 527, 530, 561, 562, 563, 577, 586, 596; 423/446; 360/102, 103, 122, 126